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AMENDED SPECIFICATION

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PATENT SPECIFICATION

Application Date: April 24, 1939. No. 12288/39. 527,951

Complete Specification Left: May 24, 1940.

Complete Specification Accepted: Oct. 18, 1940.

PROVISIONAL SPECIFICATION

Improvements in or relating to Gas-expanded Materials

We, EXPANDED RUBBER COMPANY LIMITED, a British Company, of 675, Mitcham Road, Croydon, in the County of Surrey, and ALFRED COOPER, a Swedish subject, of the Company's address, do hereby declare the nature of this invention to be as follows:—

The present invention relates to the production of gas-expanded materials from thermo-setting synthetic resins.

The term "thermo-setting synthetic resins" is to be understood to mean such compositions as harden or set by heating alone as distinct from rubber and rubber-like compositions which require a vulcanisation process with the employment of sulphur in order to convert them into the hardened condition.

The production of gas-expanded hard and soft rubber is well known in which the rubber composition in the form of dough is submitted to a gas under high pressure and then heated in order to partially vulcanise and expand the rubber. The completion of the vulcanisation and expansion of the rubber has usually been effected by heating to the requisite temperature in a mould placed in a heated press.

For the purpose of making gas-expanded thermo-setting resin compositions the above procedure is unsuitable because during the final heating and moulding the synthetic resin binder becomes liquid and unable to retain satisfactorily the gas which diffuses out and produces at most only a porous structure.

It is an object of the present invention to provide closed-cell gas-expanded synthetic resin compositions in which each cell is individually disconnected from other cells to form a very light but non-porous structure.

According to the invention a process for the production of gas-expanded material from thermo-setting synthetic resin compositions comprises partly filling a mould with the synthetic resin composition; sealing the mould to prevent any substantial escape of gas therefrom; introducing a gas into the mould under a pressure sufficient to penetrate the resinous composition; heating the mould to a temperature sufficient to cause the resin to set and before the resin becomes rigid reducing the pressure. Under these conditions during the heating for the purpose of setting the synthetic resin the gas which has penetrated into the resin composition will expand and fill up the remaining space of the partially filled mould and form a closed-cell structure owing to the fact that the composition is contained in a closed mould and while in this condition the synthetic resin will set and produce a material similar in appearance to expanded hard rubber.

Any of the well known thermo-setting resinous compositions may be employed for the purpose of the present invention, such as the phenol-formaldehyde, urea formaldehyde and alkyd resins.

In carrying out the process according to the invention a high pressure moulding frame with a suitably constructed metal gasket together with top and bottom plates is employed into which is placed the required amount of moulding powder or unset plastic material and the frame is then placed in the curing press. The frame is connected by means of a high pressure metal tube, valve and pressure gauge to a source of compressed gas. When the press is closed the gas is introduced and a pressure of 1000 to 2000 lbs. per square inch is maintained for a period depending upon the permeability of the plastic. When this period has elapsed the press is heated to a temperature sufficient to set the plastic and the gas pressure is

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released at the same time, but the mould is kept sealed until the resinous composition is set sufficiently hard to retain the gas which may be anything up to sixty minutes or even longer according to the resinous material employed. When complete setting has taken place the press is cooled and the mould opened.

Any desired gas may be used providing it is absorbed by the plastic.

Gas - expanded thermo - setting compositions of the phenol- or urea-formaldehyde type are extremely light and inert and are less water absorbent and more resistant to heat and mechanical damage than similar materials made of hard rubber. Moreover, the material is completely odourless.

The process according to this invention may also be employed for treating mixtures of synthetic resin compositions with rubber or rubber substitutes in any proportion.

Dated this 24th day of April, 1939.
For the Applicants,
F. J. CLEVELAND & COMPANY,
Chartered Patent Agents,
29, Southampton Buildings,
Chancery Lane, London, W.C.2.

COMPLETE SPECIFICATION

Improvements in or relating to Gas-expanded Materials

We, EXPANDED RUBBER COMPANY LIMITED, a British Company, of 675, Mitcham Road, Croydon, in the County of Surrey, and ALFRED COOPER, a Swedish subject, of the Company's address, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

The present invention relates to the production of gas-expanded or cellular materials from synthetic resin compositions and it is an object of the invention to produce closed-cell gas-expanded synthetic resin compositions in which each cell is individually disconnected from other cells to form a very light but non-porous structure.

The invention will be mainly described in connection with thermo-setting resins but it will be appreciated that the thermoplastic materials which only harden by cooling below the softening temperature and which are not thermo-setting, may be equally well treated by the process of the invention only in this latter case care will have to be taken to ensure that the pressure of the gas enclosed in the cells is balanced by the external pressure at all times when the material is hot, and consequently, soft, whereas in the case of thermo-setting resins the supporting pressure may be released when the material is fully set, irrespective of whether the material is cold or not.

The term " thermo-setting synthetic resins " is to be understood to mean such compositions as harden or set by heating alone as distinct from rubber and rubber-like compositions which require a vulcanisation process with the employment of sulphur in order to convert them into the hardened condition.

The production of gas-expanded hard and soft rubber is well known in which the rubber composition in the form of dough is submitted to a gas under high pressure and then heated in order to partially vulcanise and expand the rubber. The completion of the vulcanisation and expansion of the rubber has usually been effected by heating to the requisite temperature in a mould placed in a heated press.

For the purpose of making gas-expanded thermo - setting resin compositions the above procedure is unsuitable because during the final heating and moulding the synthetic resin binder becomes liquid and unable to retain satisfactorily the gas which diffuses out and produces, at most, only a porous structure.

Closed - cell gas - expanded materials other than rubber have been proposed and a heat insulation material consisting of a polymerised vinyl compound rendered highly cellular and having all the cells closed has been proposed hitherto, and the process proposed for preparing this material involves melting the material in a closed vessel containing a gas which is soluble in the substance and submitting the melted or semi-liquified mass to rapid expansion by reduction of the pressure in the melting vessel. The method suggested in this prior proposal for effecting a reduction of pressure in the melting vessel is to open a valve at the bottom of the expansion vessel so that the material is forced out and expanded either into the atmosphere or into a jacketted vessel in which a lower pressure prevails than in the melting vessel and which may be the final mould for shaping the material. In the process according to the invention about to be described, the melting and expansion take place in the mould which enables the density of the final product to be determined beforehand by adjustment of the weight of resinous material introduced into the mould and further in

the case of thermo hardening resins avoids the danger of premature setting of the resin.

According to the invention a process for the production of closed-cell gas-expanded material from thermo-plastic synthetic resin compositions comprises partly filling a mould with the synthetic resin composition; sealing the mould to prevent any substantial escape of gas therefrom; introducing a gas into the mould under a pressure sufficient to penetrate the resinous composition; heating the mould to a temperature sufficient to soften the resin and while thus softened reducing the pressure sufficiently to cause the enclosed gas to expand the material to fill the mould and thereupon hardening the material (in the case of thermo-setting resins by continuing the heating at the hardening temperature) while still supported by the mould. Under these conditions during the heating for the purpose of softening the synthetic resin the gas which has penetrated into the resin composition will expand and fill up the remaining space in the partly filled mould and form a closed-cell structure, owing to the fact that the composition is contained in a closed mould and while in this condition, if the temperature is maintained at the hardening temperature, the synthetic resin, if thermo-setting, will set and produce a material similar in appearance to expanded hard rubber.

Any of the well known thermo-setting resinous compositions may be employed for the purpose of the present invention, such as the phenol-formaldehyde, urea-formaldehyde and alkyd resins.

In carrying out the process according to the invention in the production of a thermo-hardened product a high pressure moulding frame with a suitably constructed metal gasket together with top and bottom plates is employed into which is placed the required amount of moulding powder or unset plastic material and the frame is then placed in the curing press. The frame is connected by means of a high pressure metal tube, valve and pressure gauge to a source of compressed gas. When the press is closed the gas is introduced and a pressure of 1000 to 2000 lbs. per sq. in. is maintained for a period depending upon the permeability of the plastic. When this period has elapsed the press is heated to a temperature sufficient to soften and ultimately to set the plastic and the gas pressure is released at the same time, but the mould is kept sealed and the heating is continued at the hardening temperature until the resinous composition is set sufficiently hard to retain the gas which may be anything up to sixty minutes or even longer according to the resinous material employed. When complete setting has taken place the press is cooled and the mould opened.

Any desired gas may be used providing it is absorbed by the plastic.

The various synthetic resins which have been used in commerce may be given a cellular structure by the process according to the invention but we have found that moulding powders such as the phenol-formaldehyde, urea-formaldehyde and the alkyd resin powders are only slightly expanded because the period during which these compositions are in the liquid or plastic state when heated before the material sets is too short to allow of sufficient impregnation even when using very inert gas (such as nitrogen or carbon dioxide). We have found that if a small proportion of rubber along with the necessary vulcanising ingredients is incorporated with the synthetic resin the amount of expansion is very considerably increased.

Accordingly the invention includes a process for the production of gas-expanded material from thermo-setting synthetic resin compositions as hereinbefore set out in which the synthetic resin composition is first incorporated with a small proportion, for example, 5—10% of rubber containing vulcanising ingredients.

We have found that by incorporating 5—10% of a rubber mix containing all necessary vulcanising ingredients into a phenol-formaldehyde moulding powder it was possible to lower the density of the material from approximately 40 to 50 lbs. per cu. ft. to 10 to 20 lbs. per cu. ft. The density may be even further decreased by using a greater percentage of rubber.

Using a polystyrene resin moulding powder similar results are obtained as with the phenol-formaldehyde powder and only when mixing small quantities of rubber was a suitably expanded material obtained by impregnation with nitrogen or carbon dioxide at 5,000—8,000 lbs. per sq. in. pressure. Improved expansion was obtained when using a solvent, such as ethyl chloride.

When using a poly-iso-butylene composition as sold under the Registered Trade mark "Isolene" a pressure of nitrogen of approximately 7,000 lbs. per sq. in. was employed. When gassing at atmospheric temperature very little absorption was evident and consequently very little reduction in density was obtained. As, however, the material requires a higher processing temperature under normal conditions of working it was passible to use a higher temperature in the gassing.

At a temperature of 170° C. very considerable expansion was obtained but the material did not retain the gas very well, whereas at 145° C. although a small expansion was obtained the gas was retained better. However, by including a vulcanisable rubber mixture in proportions of from 5—25%, good expansion was obtained and the material retained the gas.

In using poly-iso-butylene compositions we found it desirable to employ a compounding ingredient such as a prepared silica sold under the Registered Trade Mark "Neosyl" in quantities of not more than 25%.

The foregoing experiments show that the presence of a solvent to convert the synthetic resin into a plastic or liquid form is desirable in order to increase the amount of gas absorbed. Thus, a liquid phenol-formaldehyde resin sold under the Registered Trade Mark "Rockite" was subjected to a pressure of 5,000 lbs. per sq. in. nitrogen and maintained at 150° C. for five hours. A material having a density of 30—40 lbs. per cubic ft. was obtained and when the material was diluted with alcohol before submitting to gassing at temperatures sufficient to evaporate the alcohol and cause the resin to set an even greater amount of expansion was obtained.

It will thus be seen that in order to obtain satisfactory expansion of synthetic resin compositions it is necessary to maintain the resin in a liquid or plastic condition for a sufficient time to obtain sufficient absorption of the gas. For this reason many of the so-called casting resins are suitable for the purpose of making expanded materials. In particular, the phenol-formaldehyde resins made by condensation of phenol with an excess of formaldehyde in strongly alkaline solutions so that the condensation product is not precipitated are suitable. As is known these resins, when subsequently made slightly acid, gelatinize and by prolonged heating may be converted into hard, rigid, materials. Cellular materials may be made by treating the resin either in the liquid state before gelatinization or in the state of a gel before it is hardened.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for the production of closed cell gas-expanded material from thermoplastic synthetic resin compositions which comprises partly filling a mould with the synthetic resin composition; sealing the mould to prevent any substantial escape of gas therefrom: introducing a gas into the mould under a pressure sufficient to penetrate the resinous composition; heating the mould to a temperature sufficient to soften the resin and while thus softened reducing the pressure sufficiently to cause the enclosed gas to expand the material to fill the mould and thereupon hardening the material while still supported by the mould.

2. A process as claimed in Claim 1 wherein a proportion of rubber in the form of a vulcanisable mix is incorporated with the synthetic resin material.

3. A process as claimed in Claim 1 or Claim 2 wherein the synthetic resin material is dissolved or dispersed in a liquid vehicle.

4. A process for expanding thermoplastic materials substantially as described.

5. Expanded synthetic resin material whenever prepared by the process claimed in any one of the preceding claims.

Dated this 24th day of May, 1940.
For the Applicants,
F. J. CLEVELAND & COMPANY,
Chartered Patent Agents,
29, Southampton Buildings,
Chancery Lane, London, W.C.2.

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